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(71) Applicant *(for all designated States except US)*: CARGILL PLC [GB/GB]; Port of Tilbury, Tilbury, Essex RM18 7PU (GB).

(72) Inventors; and

(75) Inventors/Applicants *(for US only)*: CHASE, Julian, Edward [GB/GB]; 42 Genesta Road, Westcliff-on-Sea, Essex SS0 8DA (GB). SMITH, Jeffrey, Michael [GB/GB]; Flat 132, 14-16 New Crane Place, New Crane Wharf, London EC1 9TU (GB). McIVOR, Malcolm, Alfred [GB/GB]; 20 The Ridinge, Billericay, Essex CM11 2QJ (GB).

(74) Agents: COCKBAIN, Julian et al.; Frank B. Dehn & Co., Imperial House, 15-19 Kingsway, London WC2B 6UZ (GB).

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(54) Title: SPRAY DRYING

(57) Abstract

The invention relates to a process for preparing a particulate, flowable, glucose product comprising spray drying glucose syrup together with recycled dry powdered glucose to form a particulate material, wherein said particulate material is passed into a first retention zone for a first crystallisation period, then into a drying zone and subsquently into a second retention zone for a second evaporation and crystallisation period, the resulting product being milled and sifted to produce a final glucose product together with dry powdered glucose which is recycled to be spray dried. Such a process results inthe production of a high quality, free-flowing, dried glucose product of uniform consistency.

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Spray Drying

The present invention relates to a process for the preparation of a dried glucose product from glucose syrup and in particular to the preparation of such a product having an improved, uniform consistency.

Such syrup produced by the hydrolysis of various starches has been a valuable material for use in the food industry for many years. However, whilst glucose in liquid form is suitable for many applications eg. confectionery, brewing and baking, there are certain areas of the food industry in which glucose in a solid form is desirable eg. dry mixes, etc.

There are two conventional methods for obtaining glucose in a solid form from glucose syrups: slow crystallisation of pure glucose from the syrup leaving a residual mother liquor containing various impurities, and solidification of a mixture of glucose, higher sugars and impurities as a "total sugar". The first of these methods is relatively expensive in terms of capital outlay for equipment. Furthermore, it is slow in operation and a relatively large part of the glucose remains in the mother liquor which can only be sold at a low price. In addition, the final glucose product has a degree of purity well beyond that required for the food industry where the glucose product is mixed with other raw materials during use.

In view of the better economics of the second method, a number of attempts have in the past been made to realise such a method on an industrial scale. However, normal spray drying has not proved practicable due to the slow rate of crystallisation of the glucose which results in a sticky mass formed on the walls of the spray drier, and resultant downstream handling problems.

The basic spray drying procedure has been modified

by utilization of a seeded spraying technique in which syrup is sprayed over a recycled portion of the spray dried powder. The spray droplets mix with falling particles of recycled powder before falling to the bottom of the spray drier and onto the moving belt in a controlled manner. From the end of the belt the product is passed to a rotating drum and following sieving and grinding, a reasonably free flowing powder is produced, part of which may be recycled into the spraying process.

There is however the need for an improved process for the preparation of a flowable dried glucose product which is of a desired, consistent, particle size for use as a food product.

Thus, viewed from one aspect the present invention provides a process for preparing a crystalline, flowable, glucose product comprising spray drying glucose syrup together with recycled dry powdered glucose to form a particulate material, wherein said particulate material is passed into a first drying zone for a first evaporation and crystallisation period, then into a further drying zone and subsequently into a separate retention zone for a further evaporation and crystallisation period, the resulting product being milled and sifted to produce a final glucose product together with dry powdered glucose which is recycled to be spray dried.

The glucose syrup used in the process of the invention is preferably maize-derived with a glucose content (on a dry solids basis = DS) of 75 to 80% by weight and a high dextrose equivalent of between 95 and 99.

Maize-derived syrup gives rise to a product having different color, flow and fragrance than a non demineralised wheat-derived glucose.

The recycled dry powdered glucose used in the process of the invention preferably has a water content not exceeding 8% by weight and a glucose content of 94%

DSB. In normal operation it will comprise recycled particles from the milling and sifting operation subsequent to second stage retention. For process start up however any glucose powder of reasonable dryness (≤10% water content) and purity (≥90% glucose DS) may be used.

The first stage of the process involves the use of a spray drier, such as the Filtermat spray drier (from Niro A/S of Denmark) in which the moisture of the product is preferably reduced from 20-25% to 2-8% in order to provide a semi-crystalline product. In the spray drier, glucose syrup is spray dried together with recycled dry powdered glucose, e.g. in a solids weight ratio of about 2-4 to 1, preferably onto a moving surface such as a belt which then passes into a first retention zone for a short crystallisation period of, for example, 5 to 8 minutes, at 45 to 75°C. It is during this first retention period that water becomes bound into the semi-crystalline structure of the glucose product.

The sprayed product, conveniently still on the belt, then moves through a secondary drying zone which ensures the removal of any remaining free moisture from the glucose product. The product remains in this zone for 12 to 15 mins, preferably at a temperature of 45 to 60°C. Following this secondary drying process, substantially only the bound water molecules remain.

The temperature within the spray drier needs to be carefully controlled and should preferably be maintained at a temperature above the optimum temperature for crystal formation. Preferably the inlet temperature for the air in the spray drier is about 130°C and the outlet temperature is about 60-70°C, with the inlet temperature of the glucose syrup being about 70°C.

The product from the spray drier is then conveyed into a second retention zone to allow further evaporation and crystallisation to occur. Suitable

retention equipment includes a rotating drum in which the product is preferably held for a period of 15 mins to 1 hour at a temperature of 60°C.

The product discharged from the second retention zone will typically be of a particle size in the range of from 1 to 3 mm and must therefore be further milled and sifted to produce a particle size required for both the final product and for recycling to the spray drier. Such selection of an optimum particle size of 80% < 200 μm for recycling results in the production of a high quality, free-flowing dried glucose product of uniform consistency. The moisture content of the final product is preferably in the range of from 1 to 2% by weight.

The process of the invention may be carried out using conventional spraying equipment. However, in order to ensure the required final product quality, such equipment must comprise suitable retention apparatus for complete crystallisation of the product and suitable product sizing apparatus to ensure the correct size reduction of the product to form the necessary quality of recycled product.

Viewed from a further aspect the invention also provides a spray drying apparatus for performing the process of the invention, comprising means for spray drying glucose together with recycled dry powdered glucose, a rotating drum to assist crystallisation of the spray dried product, means for milling and sifting the resulting product to produce a final glucose product together with dry powdered glucose, and means for recycling the dry powdered glucose to be spray dried.

The apparatus for performing the process of the invention preferably further comprises a de-lumper located upstream of the second retention zone. Such equipment ensures that any large slabs of product emerging from the spray drier are broken up into smaller pieces prior to entering the second retention zone. In this way, the de-lumper helps to ensure the production

of particles of the size required for both the final product and for recycling to the spray drier. The de-lumper conveniently comprises a 4-bladed rotor sited within a uniformly perforated metal screen.

The means for recycling the dry powdered glucose to the spray drier conveniently comprises a conduit, eg. a conventional pipe, through which the dried glucose product travels under pneumatic pressure. Such a conduit may split into two or more, preferably about 10, sub-conduits each of which carries the recycled powder to separate high pressure nozzles located in the upper part of the spray drying chamber. The conduit conveniently splits at a point near to the top of the spray drying chamber. It has however been found that a better flow of recycled product is achieved if the conduit splits after turbulent flow in the conduit has ceased.

The process of the present invention results in the formation of a dried glucose product having superior physicochemical characteristics to those products produced by conventional spray drying techniques. In particular, the process of the invention inherently results in the formation of an amorphous product having a particularly low moisture content and a high initial water solubility at ambient temperature.

Viewed from another aspect, the invention thus provides a spray dried glucose product having an amorphous structure, a moisture content of less than 1% by weight and a solubility in water at ambient temperature of at least 55% by weight.

By "amorphous" it is meant that the spray dried product has a porous, sponge-like, high surface area structure with a degree of crystallinity $\leq$ 99% by weight.

Preferably, the spray dried product of the invention has a degree of crystallinity in the range of from 80 to 96%, more preferably 87 to 90%.

The crystallinity values referred to herein are measured by Differential Scanning Calorimetry (DSC) relative to absolute as determined by anhydrous dextrose which has a degree of crystallinity of 100% by weight.

The solubility of the spray dried product of the invention, at ambient temperature, is preferably in the range of from 55 to 63% by weight, more preferably 60 to 63%, especially 62-63%. The values given for solubility are the number of grams which dissolve in 100 grams of solution at ambient temperature. For example, 60% = 60g of product in 100g of solution.

The drying conditions used in the process of the invention moreover result in the formation of a glucose product comprising a large proportion of glucose in the β-form. Typically, the spray dried glucose product may contain up to 60% by weight of β-glucose.

The physical characteristics of the spray dried particles produced in accordance with the invention enable them to be readily compressed to form eg. tablets without the need for any further treatment and/or addition of low levels of a lubricant. For example, it has been found that both fine and coarse grades of the particles flow particularly readily into the dies of various tabletting machines, enabling the production of hard tablets of consistent weight up to speeds of 7000 tablets per minute.

The glucose product of the invention is thus of particular use in the food industry, especially in confectionery eg. in the production of hard centred sweets.

The glucose product of the invention is also of particular use in the pharmaceutical industry due to its tabletting properties which enable the ready preparation of tablet cores.

Preferred embodiments of the invention will now be described by way of example and with reference to the accompanying figures, in which:

Figure 1 schematically illustrates apparatus which represents an embodiment of the invention;

Figure 2 schematically illustrates apparatus which has a de-lumper and which represents a further embodiment of the invention;

Figures 3 and 4 are electronmicrographs (Magnification x 450 and x 4000 respectively) of spray agglomerates produced in accordance with the process of the invention;

Figure 5 is an electronmicrograph (Magnification x 300) of a spray dried dextrose monohydrate product produced by a conventional process.

Referring to Figure 1, in normal operation, glucose syrup and recycled dry powdered glucose enter the spray drying chamber 1 via inlet means 2 and 3 respectively. The feed pressure in each nozzle 19 is conveniently in the range of from 80 to 400 bar (60-90°C). A stream of hot drying air for use in the spray drying process, eg at a temperature in the range of from 140 to 175°C, enters the drying chamber 1 via inlet 4. The ratio by weight of feed material to recycled material entering the drying chamber is preferably between 2 to 4:1.

Drying air leaves the drying chamber 1 through multiple ducts 5 and any powder particles are separated from the air in cyclones 6. The drying air leaves the apparatus through a stack 15.

The spray dried product is collected as a porous cake on moving belt 7 which passes through a first retention zone 8 and subsequently through a secondary drying zone 9. The dried product then passes through a cooling zone 16 prior to being conveyed into rotating drum 10 for further evaporation and crystallisation. Drum 10 is preferably provided with hot air at an inlet temperature in the range of from 80 to 120°C. The air exits the drum at a temperature of from 50 to 90°C and is filtered and exhausted to the atmosphere. The resulting product leaves the drum with a moisture

content of from 0.5 to 1.0% by weight and is then pneumatically conveyed to a holding bin 20.

The resulting product is then passed through sifter 11 followed by grinder 12 to produce particles of a size required for both the final product 13 and for recycling to the spray drying chamber 1. Fine grade product passes into bin 17 and coarse grade product into bin 18. Each product is then bagged.

The particles for recycling are passed back to the spray drying chamber via pipe 14.

Figure 2 illustrates a preferred embodiment of the invention in which the dried product is passed through a de-lumper 21 prior to entering rotating drum 10. A second de-lumper may also be provided downstream of the sifter 11.

Figures 3 to 5 attached hereto illustrate the amorphous nature of the glucose product of the invention compared to the crystalline structure of dextrose monohydrate prepared by a conventional spray drying process. Table 1 below compares the product of the invention with dextrose monohydrate and sucrose.

Table 1

	Glucose product of the invention	Dextrose	Sucrose
Chemical Analysis (% by weight)	94% dextrose 3% maltose 3% higher sugars	>99.5% dextrose	>99.9% sucrose
Crystallinity measured by DSC = Differential Scanning Calorimetry (% by weight)	87-96%	100%	100%
Forms of α/β dextrose measured by polarimetry (% by weight)	60% β	100% α	*
Moisture content (% by weight)	<1%	8-9% <2% (anhydrous)	<0.02%
Maximum solubility in water at ambient temperature (% by weight)	62-63%	55%	67-68%

* Sucrose is a disaccharide and is thus not comparable

Claims:

1. A process for preparing a particulate, flowable, glucose product comprising spray drying glucose syrup together with recycled dry powdered glucose to form a particulate material, wherein said particulate material is passed into a first retention zone for a first crystallisation period, then into a drying zone and subsequently into a second retention zone for a second evaporation and crystallisation period, the resulting product being milled and sifted to produce a final glucose product together with dry powdered glucose which is recycled to be spray dried.

2. A process as claimed in claim 1 wherein said glucose syrup is maize-derived with a glucose content (DS) of 75 to 80% by weight and a dextrose equivalent of between 95 and 99.

3. A process as claimed in claim 1 or claim 2 wherein said particulate material has a water content of between 1 and 2% by weight.

4. A process as claimed in any one of claims 1 to 3 wherein said first crystallisation period is in the range of from 5 to 8 minutes.

5. A process as claimed in any preceding claim wherein said second retention zone comprises a rotating drum.

6. A process as claimed in any preceding claim wherein said second crystallisation period is in the range of from 15 minutes to 1 hour.

7. A glucose product produced by a process as claimed in any preceding claim.

8. Apparatus for performing a process as claimed in any one of claims 1 to 6 comprising means for spray drying glucose together with recycled dry powdered glucose, a rotating drum to assist crystallisation of the spray dried product, means for milling and sifting the resulting product to produce a final glucose product together with dry powdered glucose, and means for recycling the dry powdered glucose to be spray dried.

9. Apparatus as claimed in claim 8 further comprising a de-lumper located upstream of the rotating drum.

10. Apparatus as claimed in claim 8 or claim 9 wherein said means for recycling the dry powdered glucose comprises a conduit which splits into a plurality of sub-conduits each of which carries the powdered glucose into the means for spray drying.

11. Apparatus as claimed in claim 10 wherein said conduit splits after turbulent flow has ceased.

12. A spray dried glucose product having an amorphous structure, a moisture content of less than 1% by weight and a solubility in water at ambient temperature of at least 55% by weight.

13. The use of a glucose product as defined in claim 12 in the production of compressed products.

14. Use as claimed in claim 13 in the production of pharmaceuticals or confectionery.

FIG.1

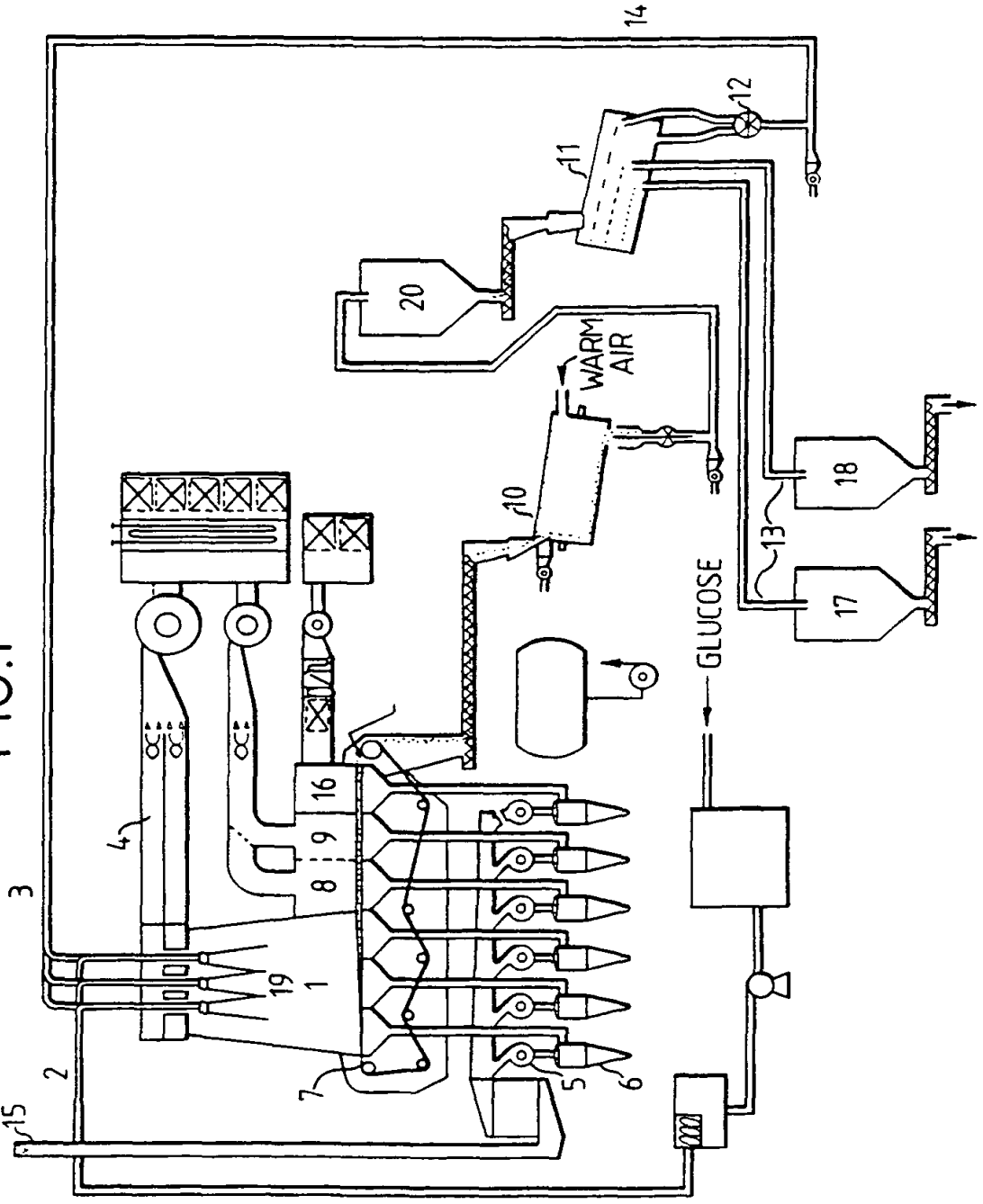

FIG. 2

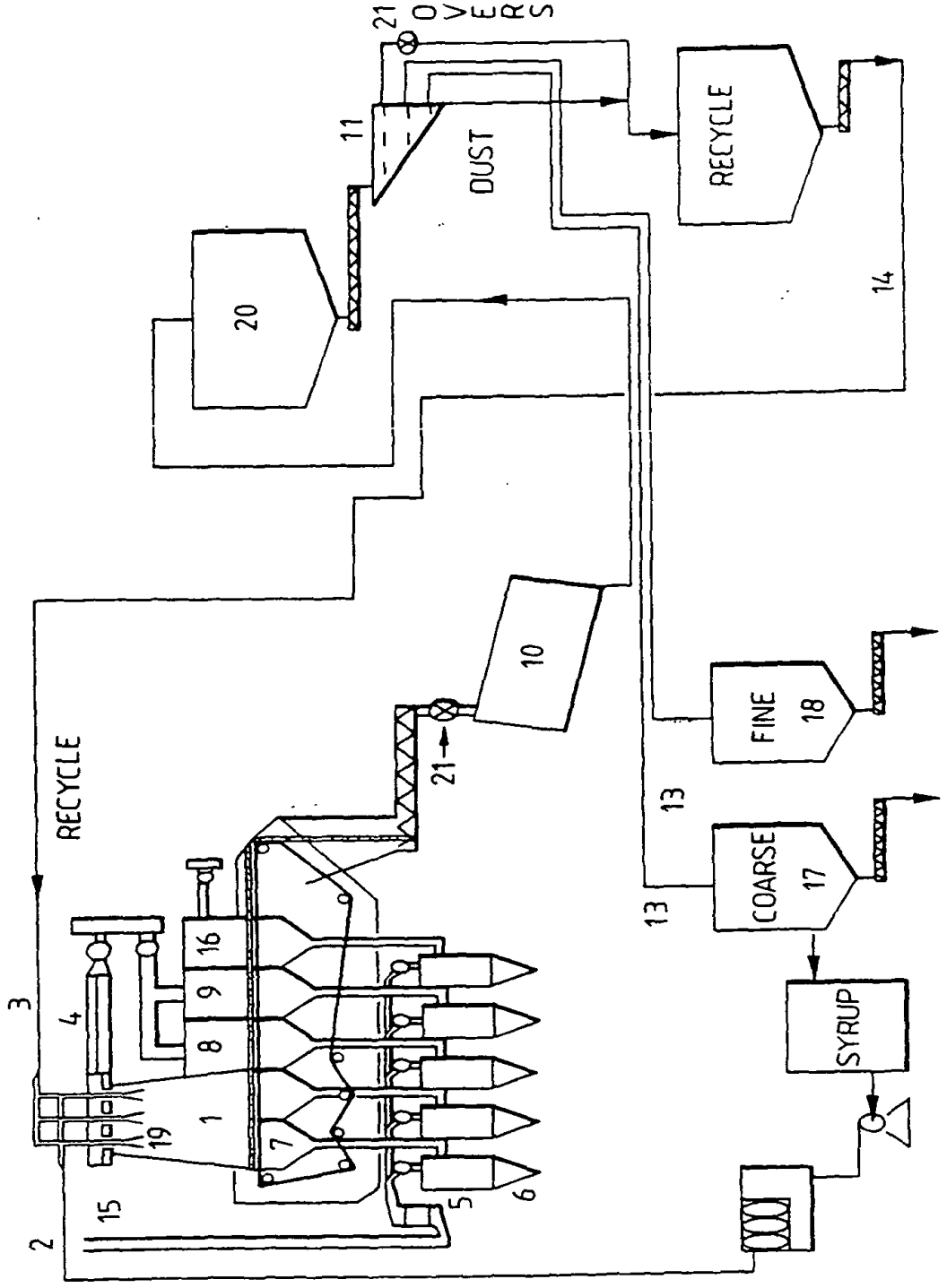

FIG. 3

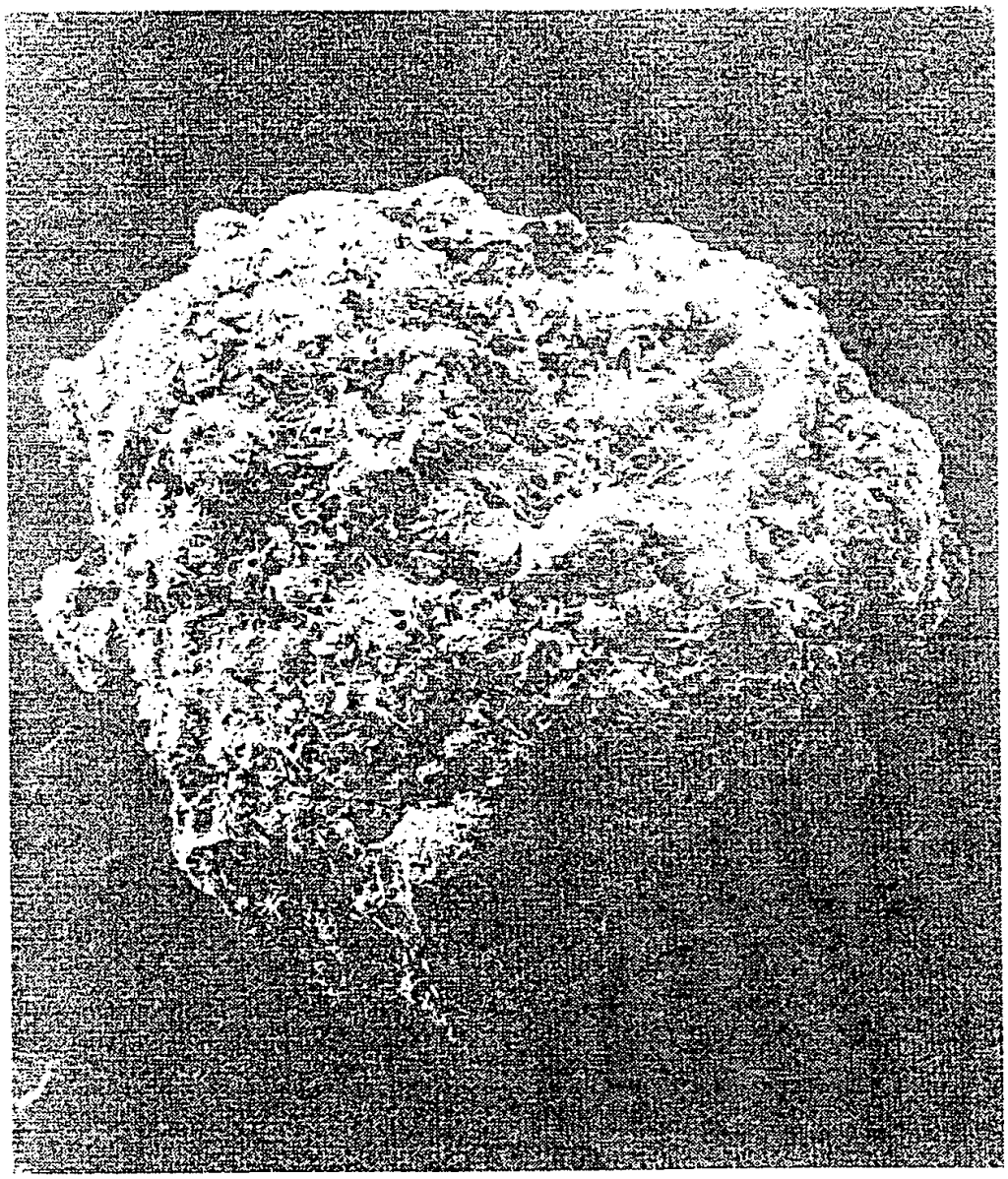

FIG. 4

FIG. 5

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(54) Title: SPRAY DRYING

(57) Abstract

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A. CLASSIFICATION OF SUBJECT MATTER
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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	BE,A,441 172 (GLUCOSERIES REUNIES) 31 May 1941 see claims; figure see page 2, line 18-33 see page 3, line 10-14 see page 4, line 13-16	1-3,7,8, 12-14
A	GB,A,2 132 495 (CPC INTERNATIONAL) 11 July 1984 see the whole document	1-14

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Date of the actual completion of the international search	Date of mailing of the international search report
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BE-A-441172		NONE	
GB-A-2132495	11-07-84	AU-A- 2221283	21-06-84
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		JP-A- 59166100	19-09-84